

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Docket No: Q87635

Toshihiko OKAMOTO, et al.

Appln. No.: 10/ 533,309 Group Art Unit: 1796

Filed : January 5, 2006 Examiner: Robert S. LOEWE

Title : CURABLE COMPOSITION AND METHODS FOR IMPROVING
RECOVERY PROPERTIES AND CREEP PROPERTIES

DECLARATION UNDER RULE 132

Honorable Commissioner of Patents and Trademarks,
Alexandria, Virginia 22313-1450

Sir:

I, Toshihiko Okamoto, a citizen of Japan and having postal mailing address of 1-chome 19-19, Nonoue, Akashi, Hyogo 673-0017, Japan, declare and say that:

In March 1994, I was graduated from Graduate School of Engineering, Osaka University, and received a master's degree in the field of chemistry;

Since April 1994, I have been employed by Kaneka Corporation and engaged in the work of research and development of modified silicone composition for sealing material in High Performance Polymers Division;

I am an inventor of the above-identified application and am familiar with the technical field of the present invention;

I respectfully submit herewith my exact report;

In order to show the excellent properties of the composition of the instant claims, I have carried out the following experiments. For easy comparison between the

results of experiments, the experiments below were performed under a condition where the added amounts of tin carboxylate were identical, and carboxylic acid ingredients were not used in all the following experiments.

Procedure of Experiments

The curable compositions listed in the table below were prepared in the same manner as Examples 1-4 and Comparative Examples 1 and 2 (Table 1) of the instant specification. Skin formation time, recovery ratio, and creep resistance of each composition were determined in the manner as described below.

The organic polymer (A-2) in the Table below corresponds to the organic polymer obtained in Synthetic Example 2, and the polymer (A-4) corresponds to the organic polymer obtained in Synthetic Example 4.

<Skin formation time>

The skin formation time of each curable composition was evaluated in the following manner:

A composition was filled in an about 5-mm thick molding frame with a spatula, and thinly spread with the spatula in a planar shape. The time at which the spreading was completed was defined as the time of start of curing. The surface of the composition was then touched with a spatula, and the time at which the composition did not adhere to the spatula any longer was determined. The period from the start of curing to the time at which the composition did not adhere to the spatula any longer was defined as the skin formation time. The skin formation time was measured in a condition at 23°C, 50%RH.

<Recovery ratio (1)>

Each of the compositions listed in the Table below was aged at 23°C for 3 days and then at 50°C for 4 days to prepare sheets which were about 3 mm in thickness. These sheets were stamped out by No. 3 dumbbell-shaped dies, and the dumbbell specimens were fixed for 24 hours in an oven at 60°C in a state in which 20 mm token line intervals were stretched to 40 mm (100% elongation). These dumbbell specimens were released at 23°C, whereby the recovery ratio (1) was determined from the ratio that the token line had recovered after 1 hour. A greater recovery ratio indicated superior recovery properties.

<Recovery ratio (2)>

Each of the compositions listed in the Table below was aged at 23°C for 3 days and then at 50°C for 4 days to prepare sheets which were about 3 mm in thickness. These sheets were stamped out by No. 3 dumbbell-shaped dies, and the dumbbell specimens were fixed for 2 hours in an oven at 60°C/95%RH in a state in which 20 mm token line intervals were stretched to 40 mm (100% elongation). These dumbbell specimens were released at 23°C, whereby the recovery ratio (2) was determined from the ratio that the token line had recovered after 1 hour.

<Creep resistance>

Each of the compositions listed in the Table below was aged at 23°C for 3 days at 50°C for 4 days to prepare sheets which were about 3 mm in thickness. These sheets were stamped out by No. 3 dumbbell-shaped dies, and the dumbbell specimens were marked with a token line at 20 mm intervals. One terminal of this dumbbell specimen was fixed in an oven at 60°C/95%RH, whereby the dumbbell specimen was made to hang down. A 0.2-MPa force was loaded onto the lower terminal of the hanging-down

dumbbell specimen. The displacement difference of the token line interval distances between immediately after the force was loaded and 200 hours after the force was loaded was measured. A smaller displacement difference indicated superior creep resistance.

Table

Composition (Part(s) by weight)			Experiments					
			1	2	3	4	5	6
Polyoxy-alkylene polymer	A-2 <Component (A1)>	Trimethoxysilyl group	100	100	100	100		100
	A-4	Methyldimethoxysilyl group					100	
Filler		HAKUENKA CCR	120	120	120	120	120	120
		Tipaque R-820	20	20	20	20	20	20
Plasticizer		DIDP	55	55	55	55	55	55
Thixotropy-imparting agent		Disparlon #6500	2	2	2	2	2	2
Photostabilizer		Sanol LS770	1	1	1	1	1	1
Ultraviolet Absorber		Tinuvin 327	1	1	1	1	1	1
Antioxidant		Nocrac SP	1	1	1	1	1	1
Adhesion-imparting agent		A-1120	3	3	3	3	3	3
Si compound	Silicate <Component (B)>	Methyl Silicate 51 (Condensate)	2				2	2
		Ethyl Silicate 40 (Condensate)		2				
		Ethyl Silicate 28 (monomeric)			2			
	Vinyltrialkoxysilane	A-171				2		
Curing catalyst	Tin neodecanoate <Component (C)> <a tin neodecanoate having a quaternary α -carbon atom>	Neostann U-50	3.4	3.4	3.4	3.4	3.4	
	Tin carboxylate <a tin carboxylate having a tertiary α -carbon atom>	Neostann U-28						3.4
	Amine	Laurylamine	0.75	0.75	0.75	0.75	0.75	0.75
Skin formation time		min.	50	50	50	55	70	90
Recovery Ratio (1)		%	92	91	89	83	75	92
Recovery Ratio (2)		%	78	78	74	71	70	77
Creep Resistance <Displacement>		mm	4.0	4.3	5.5	7.3	18.1	3.9

[Results]

The compositions of Experiment Nos. 1 to 3 were compositions which fall within the scope of the amended claims of the instant application. The compositions of Experiments No. 1 to 3 showed high recovery ratios, excellent creep resistance (displacements were small), and short skin formation times (curability was excellent).

The composition of the Experiment No. 4 contained a Si compound other than silicates. Such a composition was inferior to the compositions of Experiment Nos. 1 to 3 in recovery ratio and curability. The difference in recovery ratios between the Experiment No. 4 and the Experiment Nos. 1-3 were seemingly small, but creep resistance, which is more suitable index as practical evaluation, was significantly different therebetween. A composition with high creep resistance is suitable particular in an application which is sensitive to a shearing caused by deformation of adhesive layers.

The composition of the Experiment No. 5 contained an organic polymer having a methyldimethoxysilyl group. Such a composition was poorer in curability, recovery ratio, and creep resistance than the composition of Experiment No. 1.

The composition of the Experiment No. 6 contained a tin carboxylate having a tertiary α -carbon atom. Such a composition was remarkably poorer in curability than the composition of Experiment No. 1.

As evident from the above results, a composition with excellent recovery property, creep resistance, and curability was obtained only in the case where the (A1) component, a polyoxyalkylene polymer having a trimethoxysilyl or triethoxysilyl group, the (B) component, a certain specific Si compound (a silicate), and the (C) component, a certain specific curing catalyst (tin neodecanoate) were combined together.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Signed this 22nd day of October, 2010

Toshihiko Okamoto

Toshihiko Okamoto